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Electron-beam guns hit the mark

NASA's Byron Lichtenberg loads a crystal-growth experiment aboard a mock-up of Spacelab

INTERNATIONAL MAGAZINE OF RESEARCH AND DEVELOPMENT

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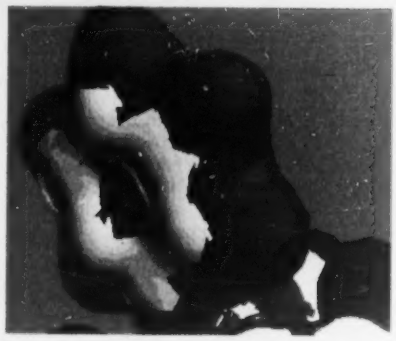

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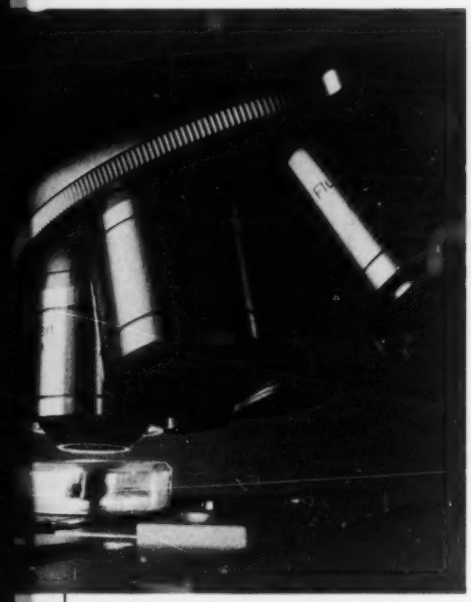
ABOUT THE COVER

NASA's Byron Lichtenberg loads an experiment aboard a Spacelab mock-up. NASA plans to launch Spacelab aboard Columbia in December. Critics, however, fear that NASA's mission for the 1990s already is off course. See story on page 42. Photograph by Neil MacDonald.



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BUSINESS

Defense R&D spending shoots up in 1st quarter

AFTER RISING a meager 0.4% in the last quarter of 1989, defense expenditures for contractual research and development rose 8.7% in the first quarter of 1990 above what was spent in first-quarter 1989.

This amounts to a seasonally adjusted annual figure of $32.4 billion, or 5.2% above 1989's total spending of $30.8 billion.

National defense purchases amounted to an annualized $309 billion in the first quarter of 1990—up 3.5% over a year ago. When adjusted for inflation, purchases actually rose only 0.6%. This rate accounted for fewer government purchases of some types of military equipment.

According to the Electronic Industries Assn., the budget for research, development, test, and evaluation (estimated at about 90% of the total defense R&D budget) will decline from $39.3 billion in 1988 to $28.8 billion in 1999 (measured in FY90 dollars).

—Julie Handelman

Japan wins larger piece of U.S. aerospace pie

THE JAPANESE aerospace industry's 10% share of Seattle-based Boeing's 777 program is the largest share of any U.S. aircraft development ever captured by an "outside" company.

The 777 program is a twin-engined, long-range aircraft capable of carrying 350 passengers. The advanced design is expected to incorporate composite structural components, fiber-optic cables, and integrated avionics.

However, the Japanese firms, Mitsubishi, Kawasaki, and Fuji Heavy Industries, felt the pinch of development costs when they were unable to meet the 25% equity partnership requirements that they initially wanted.

For a 25% equity share, Boeing required at least $1 billion of the $4 billion estimated development cost. The Japanese firms, already heavily committed in money and staffing to developing the FS-X fighter, didn't have the $1 billion or the additional engineers to support a 25% equity share.

Mitsubishi also recently announced a joint materials, aircraft engine, and aerodynamics research program with Germany's Daimler-Benz AG.

Total Japanese involvement in the 777 is expected to go beyond the Boeing-Mitsubishi-Kawasaki-Fuji arrangement. Three Japanese aircraft engine companies are bidding on engines for the 777. Toray Industries, a supplier of carbon composites, and the joint venture of Dublin, CA-based Hexcel and Dainippon Ink & Chemicals, a supplier of honeycomb and advanced composite panels, are potential suppliers of composite structures.

Other Japanese companies that already supply parts for Boeing's existing 767 program would be expected to get follow-on contracts. *—Tim Studt*

Moderate growth seen for x-ray fluorescence

CONTINUING technical innovations in x-ray fluorescence instruments will increase this market at an annual rate of more than 11% to $440 million worldwide in 1993, according to Strategic Directions Intl., a Los Angeles-based analytical-instrument consulting firm.

This total includes wavelength- and energy-dispersive x-ray fluorescence and x-ray microanalysis accessories used on electron microscopes and microprobes.

Top 1991 R&D spenders are a rerun of 1990

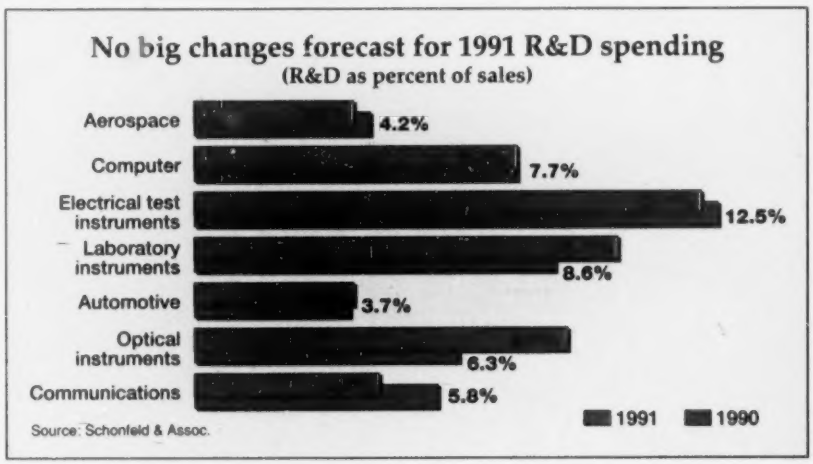
THE TOP 10 corporate R&D spenders next year will be the same companies who were the top 10 spenders in the world in 1990, according to a study by Schonfeld & Associates, an Evanston, IL-based management consulting firm.

The ranking of the top five spenders won't change at all, with General Motors ($5.975 billion) barely edging out IBM ($5.969 billion). Hitachi, Matsushita, and Ford Motor round out the top five, followed by AT&T, NT&T, NEC, Toshiba, and Philips NV.

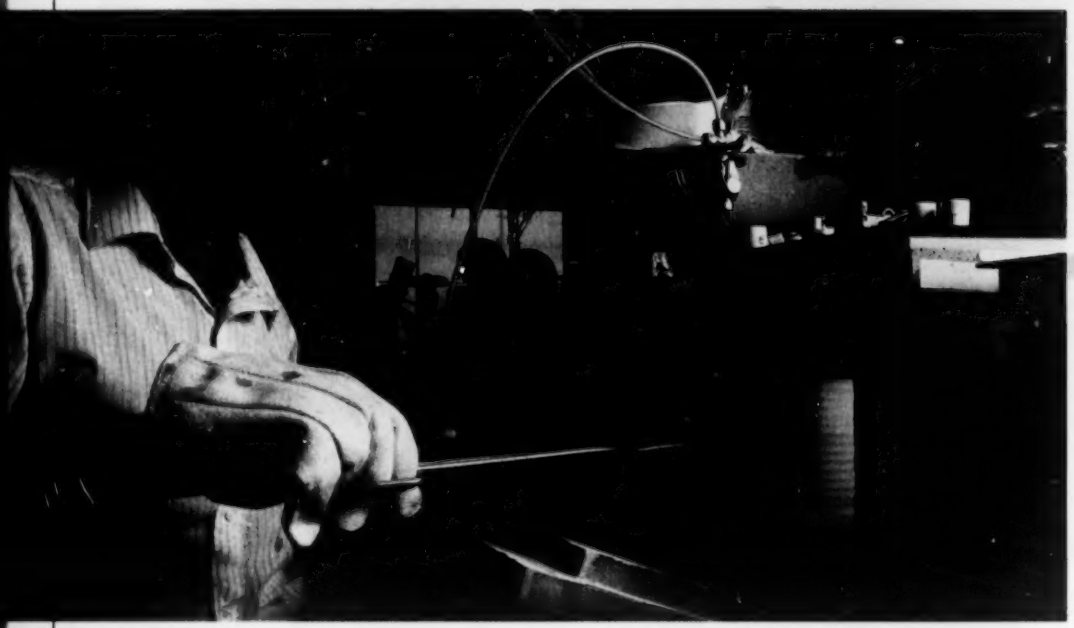
The computer industry is forecast to be the biggest R&D spender in 1991 at $21.7 billion, up 9.7% over R&D spending in 1990.

Aerospace R&D is starting to show some effects of the defense slowdown, with the biggest spender, United Technologies, forecast to increase its R&D spending by only 4% to $1 billion.

For a copy of *R&D Ratios & Budgets*, ($295): 708/869-5556. □



***Changes in R&D spending** forecast for 1991 reflect lower defense spending, a maturing of the computer industry, and increased use of optical instruments in quality control.*



***Chemist** Ed Nazzal of IBM's Almaden Research Center, San Jose, CA, heats a sample of oxides to turn it into a ceramic superconductor. The U.S. has a formidable HTS research program in place, but a report by the U.S. Office of Technology Assessment questions whether industry is in HTS for the long haul to commercialization.*
Photo: IBM/Don Kenney

SUPERCONDUCTIVITY

Is U.S. vulnerable in HTS race?

HIGH-TEMPERATURE superconductivity (HTS), one of the most exciting scientific developments in the 1980s, will be the most challenging test to U.S. competitiveness in the 1990s, according to a report by the U.S. Office of Technology Assessment (OTA).

While the government agency believes that the U.S. has responded well to the early challenge, it also questions whether U.S. companies are in a position to go toe-to-toe with the Europeans and the Japanese in HTS commercialization.

"There is a serious question whether U.S. industry, as it is currently financed and managed, can compete in markets for these technologies in the next century," the report states. "HTS is a test case . . . of [U.S.] ability to look beyond the immediate future and sustain a consistent R&D effort over the long term."

The U.S. and Japan each spend about $220 million a year on superconductivity research. But in Japan the funding comes more from large companies and less from the government. In the U.S., the opposite is true.

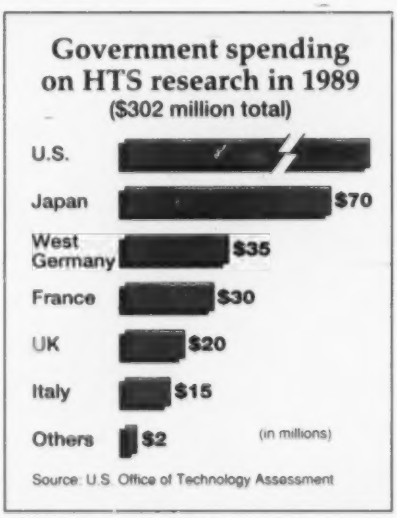
This makes the U.S. program susceptible to budget cutbacks and changing priorities.

The Dept. of Defense is the largest supporter of HTS research, but the thaw in the Cold War could shrink this funding source.

"Government funding, especially through the Dept. of Defense, is not the most direct route to commercialization," says Bill Gallagher, manager of exploratory cryogenics at IBM Corp., Yorktown Heights, NY.

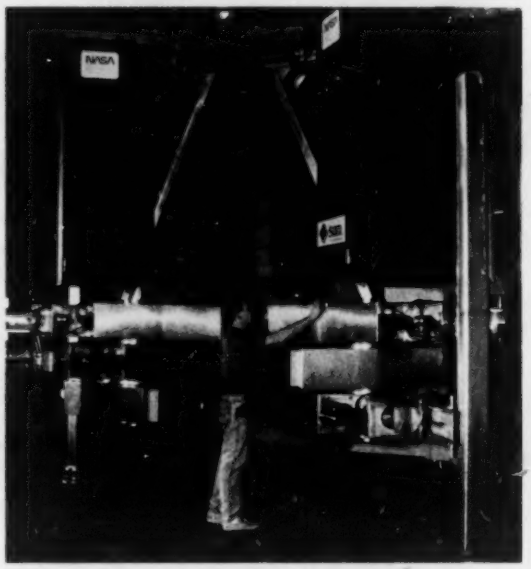
What is needed are more U.S. corporations to fund HTS research over the long term, a bolstering of university research, and coordination of the HTS effort at the national level, OTA states.

HTS products should begin coming to market in five years, but the significant products are 10 to 20 years away, says OTA. This is much longer than



***The U.S. government** outspends Japan on HTS research by nearly 2-to-1, but Japanese companies are in a better position for HTS commercialization, states the U.S. Office of Technology Assessment.*

Autonomous robot is ready to scale new heights



A researcher at Carnegie Mellon Univ., Pittsburgh, adjusts Ambler (autonomous mobile exploration robot), which is being developed for NASA for possible planetary and lunar exploration missions. A final version of Ambler will have a laser scanner for visual perception and an on-board computer for reasoning.

Ambler, with its 3-m-long (10-ft) legs, will be able to step over 1-m-dia obstacles on a 30-deg slope while keeping its body level. Although the robot is designed to traverse rough terrain, it will do so at a slow pace, covering about 1 km per day.

many large U.S. companies are willing to invest in a research program.

Small start-up firms need a large funding base to make it through the lean early years. There is concern in industry as to whether the small companies can survive living off their early products.

But for small companies the question is: What are the big U.S. corporations doing?

"The situation is competitive right now," says Gregory Yurek, president and CEO of American Superconductor Corp., Watertown, MA. "If we don't see the continued funding from the government and we don't see the large U.S. companies getting more involved, then we will start losing the competition."

Some say for the U.S. to succeed in HTS, the U.S. needs an industrial policy, an idea the Bush administration has opposed.

"I have some sympathy for those against an industrial policy," says John Hulm, chief scientist emeritus, Westinghouse Electric Corp., Pittsburgh. "But in the modern world, where other countries' governments are interfering, we probably need to do some interfering too."

"This really is a case of the U.S. vs. the U.S.," adds Ed Mead, manager of business development for superconductivity at DuPont Co., Wilmington, DE. "Are we going to be fat and sloppy and not move quickly or very well, or are we going to move assuredly?" —*Skip Derra*

OCEANOGRAPHY

Iron is seen as tonic for global warming

OCEANOGRAPHER John Martin says the simplest way to combat global warming is to pump some iron. Actually, to pump about 300,000 tons of iron into what amounts to 18% of the world's oceans.

The iron "fertilizer" would help phytoplankton in the ocean bloom, which in turn would absorb as much as 2 billion tons/yr of CO_2, according to Martin's estimates. This would put a big dent into the 3 billion tons/yr of CO_2 buildup in the atmosphere.

"It could have a huge effect," says Martin, director of Moss Landing (CA) Marine Laboratories.

Giving iron-poor oceans "a shot of Geritol" has been a pet idea of Martin's for two years now. "All you have to do is add a pinch of iron here and there, and the plants will bloom," he says.

But Martin is quick to add that iron fertilizing is only an idea. A lot of work still has to be done to explore fertilization sites and assess environmental effects.

"Iron fertilizing certainly is one way to increase the fixation of carbon in the oceans," says Roger Revelle, former director of the Scripps Institution of Oceanography, La Jolla, CA. "But the iron would have to be spread over an enormous area. Nobody knows how to do that." □

SPECTROSCOPY

Researchers aim new detector at the heart of nuclear matter

BRITISH AND FRENCH scientists are building the world's most sensitive gamma-ray detector.

An $8.5 million agreement between the UK Science & Engineering Research Council's Daresbury Laboratory, Warrington, and the French National Institute of Nuclear Physics and Particle Physics, Paris, calls for development, construction, and utilization of the Eurogam gamma-ray detector.

Eurogam is expected to be two orders of magnitude more sensitive than any other gamma-ray detector.

It will be used to study some of the most exotic forms of nuclear matter, including the super-deformed nuclear pulsars.

Eurogam, in addition to studying the limits of nuclei stability, will be novel in that it will be shipped between sites in England and France. Eurogam will be located at Daresbury for the first year after it is completed, and the 45-detector array will be connected to Daresbury accelerators and a recoil mass spectrometer for nuclear structure studies.

By its second year of operation, Eurogam will be built up to a 70-detector system. Then it will be moved to the French Vivitron accelerator, now under construction in Strasbourg. □

NONDESTRUCTIVE EVALUATION

Researchers adapt NDE systems for thick composites

AS INDUSTRY BEGINS to use composite parts in primary structural applications, there is a growing need to develop nondestructive evaluation (NDE) methods to inspect the parts.

Current NDE methods are useful in inspecting thin composites, but researchers are finding new challenges in developing systems for thick composites, says Glenn Light, manager of the NDE Assessment Section, Southwest Research Institute, San Antonio.

Primary structural applications of

Surface Science Pushes Theoretical Limits

R&D Perspectives

New microscopy and surface analysis instruments help you probe the composition and structure of materials.

By ***Tim Studt,*** *Associate Editor,* R&D Magazine

MICROSCOPY has advanced further in the past 10 years than it had in all the previous 300 years, since Antonie van Leeuwenhoek invented the compound optical microscope.

The past decade saw the development of microscopes that image individual atoms, microscopes that can peer inside a living cell without damaging the cell, and microscopes that let you watch crystals grow at 1,000 C.

Even Leeuwenhoek's basic light microscope (LM) has seen evolutionary breakthroughs with electronic imaging and video enhancements that improve its resolution and allow you to manipulate images.

Research, such as that in near-field scanning techniques being performed at Cornell Univ., Ithaca, NY, also is bringing LMs into the performance realm of electron microscopes, says Ernst Keller, LM technical manager for Carl Zeiss Inc., Thornwood, NY.

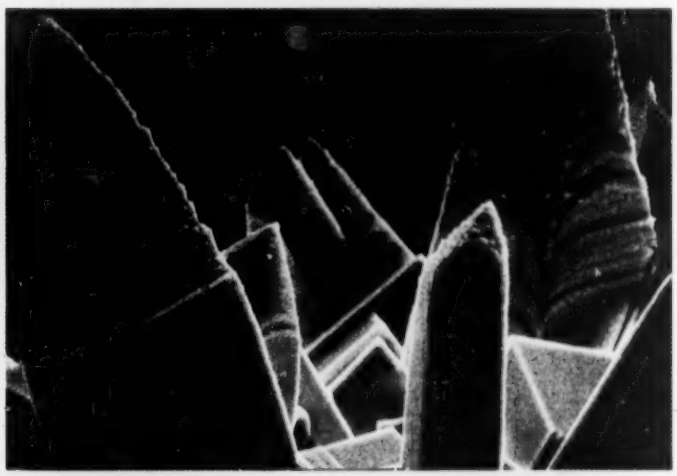
The most significant event in microscopy is the availability of powerful and cost-effective electronic systems. By adding electronic imaging and analysis capabilities to a microscopy system, you can see and use more of the image information. None of the electron microscopes and scanning probe microscopes would have been possible without the development of electronic imaging systems.

Four hot microscopes

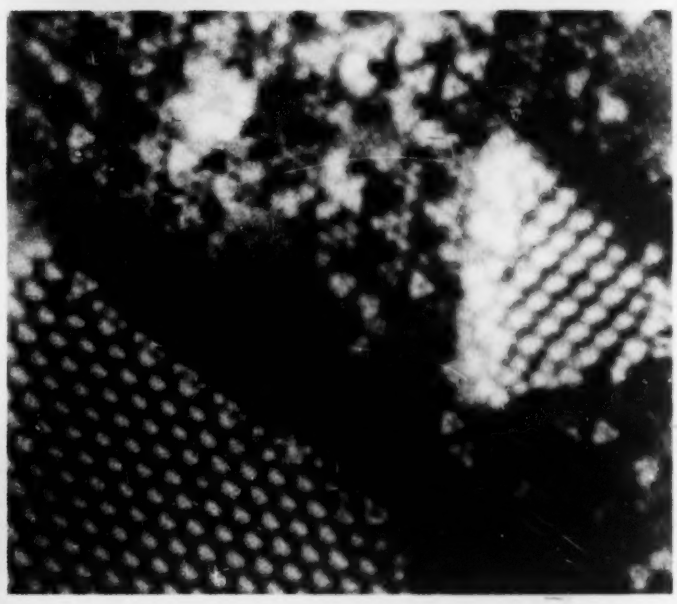
Four microscopy systems giving you imaging capabilities that until now were not possible are confocal scanning microscopes, scanning tunneling microscopes, scanning force microscopes, and environmental scanning electron microscopes.

Confocal scanning microscopes: These microscopes let you generate 3-D images of a sample, both external and in-

Scientist at Univ. of Oxford, England, uses a scanning proton microprobe to examine an IC function. The SPM can detect low-intensity x rays from trace elements because no background x rays obscure the signal.



***These KCl** crystals were condensed from gas at 600 C. An environmental scanning electron microscope allows you to observe their growth.*



***Three material** phases (lower left, center, and upper right) of an Sn-induced reconstruction in a Si(111) surface are revealed in this UHV STM image. Image by Park Scientific Instruments.*

You can literally 'walk' a confocal microscope through a sample as you image at different focal planes in it.

Perspectives

ternal features, without cutting or damaging the sample.

With a confocal microscope you focus your optics at one plane of a sample, electronically store the resultant image, move the focus down a few micrometers into the sample, and take another image. You can literally "walk" a confocal microscope through a sample as you image at different focal planes in it.

To construct a solid model, you then use a software package to recombine the images. Some models use up to 150 image slices.

The confocal concept relies on the focusing of a light source onto a sample through high numerical aperture lenses. This causes the light to approach the sample at angles that produce a thin plane of focus.

Light reflected from the sample passes back through the optic system, which focuses the beam through a pinhole aperture and onto a detector. Light reflected from above or below the plane of focus is blocked at the pinhole from reaching the detector, creating a high-resolution image with high contrast.

Confocal scanning microscopes are available with tungsten filaments or laser light sources. A tungsten source lets you image color samples, while the high intensity of a laser source provides images with high resolution and contrast.

Confocal laser scanning microscopes (CLSM) are especially useful in the imaging of delicate biological samples, such as those that can be stained with fluorescent dyes.

What's the difference?

SINCE MANY surface science techniques operate in microscope or microprobe (surface analysis) modes, when does a microscope stop being just a microscope and when does it become a microprobe?

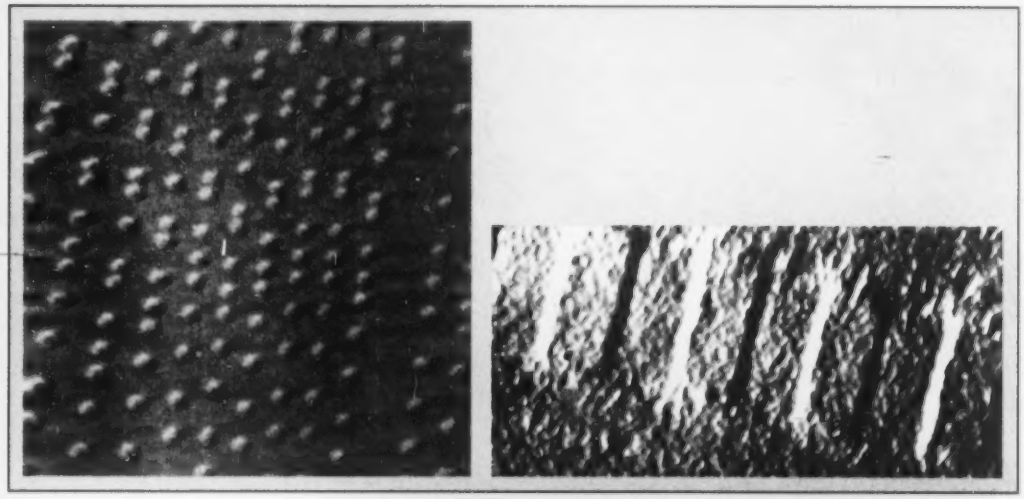
"Microscopes only show you the morphology of a surface," says Charles Evans, president of Charles Evans & Associates. "By themselves they do not identify elements or molecules."

A microprobe provides information that's used to "tag" a constituent. In the process of obtaining this information, you also might be able to image the surface.

Microscopes: From macroviews to atomic views

	Acronym	Magnification range, times	Resolution, nm	Analysis time*, sec	NDT Yes/No	Operating voltage, eV	Environment, Vacuum/ Ambient	Approximate equipment cost (U.S. $)
Light microscope	LM	5 to 2,000	200	10	Yes	0	Ambient	$2,000 to $100,000
Scanning electron microscope	SEM	10,000 to 250,000	5	60	Yes‡	500 to 40,000	Vacuum/ Ambient†	$40,000 to $400,000
Scanning electron microscope/Field emitter	SEM/ FE	10,000 to 400,000	1.0	90	Yes‡	100 to 300,000	Vacuum	$200,000 to $600,000
Transmission electron microscope	TEM	1,000 to 1,000,000	0.15	300	No	30,000 to 1,000,000	Vacuum	$100,000 to $5,000,000
Scanning transmission electron microscope	STEM	100,000 to 1,000,000	0.5	300	No	1,000 to 50,000	Vacuum	$200,000 to $800,000
Scanning TEM with selective area diffraction	STEM/ SAD	1,000 to 1,000,000	0.15	30	No	30,000 to 1,000,000	Vacuum	$100,000 to $5,000,000
Scanning laser acoustic microscope	SLAM	10 to 500	1,000	60	Yes	0	Ambient	$600,000 to $800,000
Confocal scanning microscope	CSM	100 to 10,000	0.1	20	Yes	0	Ambient	$50,000 to $200,000
Scanning tunneling microscope	STM	5,000 to 10,000,000	0.0001	300	Yes£	0 to 1 V	Vacuum/ Ambient	$30,000 to $200,000
Atomic force microscope	AFM	5,000 to 2,000,000	0.0005	300	Yes	0	Ambient	$75,000 to $200,000

* Time listed is to acquire an image with no analysis required.
† Most SEMs are kept under vacuum, all measurements are made in vacuum.
‡ Sample preparation may be destructive.
£ STMs are generally nondestructive, but its probe beam may do damage.



Magnetization *patterns of longitudinal (near left) and magneto-optic recording media (far left) are revealed with a magnetic force microscope by researchers at IBM. Both pictures have the same scales—each longitudinal bit is about 5-μm wide.*

"CLSMs are just starting to be used in materials science analyses," says Barbara Foster, technical manager for Sarastro Inc., Philadelphia. "With the CLSM, we've imaged several millimeters in depth into styrofoam and have focused through thin films of copper."

Scanning tunneling microscopes: It has been eight years since IBM researchers in Zurich, Switzerland, built a scanning tunneling microscope and used it to reveal the first images of individual atoms. Today, commercial versions of STMs are available and, except for special analysis requirements, buying a commercial instrument is more practical and less expensive than building your own system.

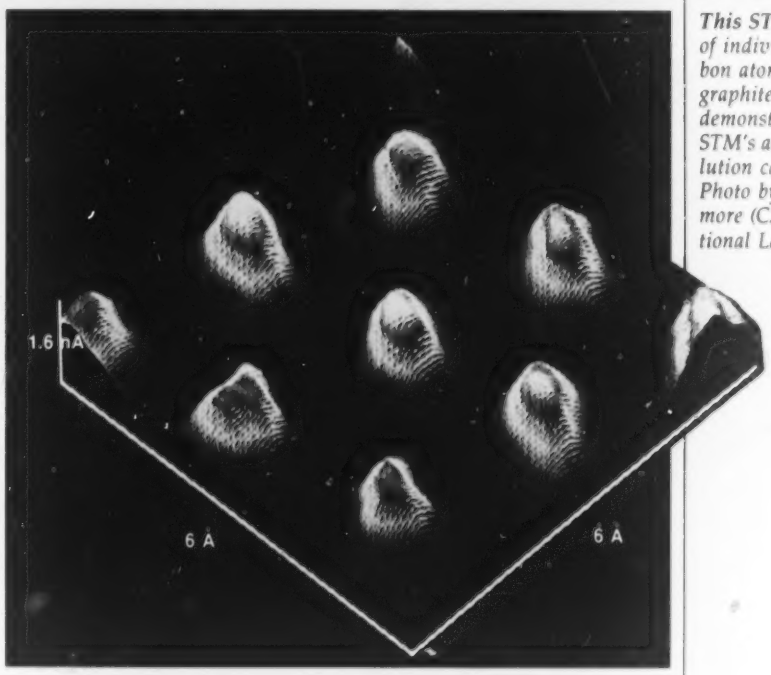
The commercial instruments include graphic workstations, software, and electron-tunneling spectroscopy tools.

Because STMs have become relatively inexpensive, they have become fairly common in labs. This has resulted in numerous journal papers that refer to STM analyses and further establish its credibility.

Still, the STM technique is not without its problems.

"STMs are a number of years away from being a tool that can be used off the shelf by anybody," says Rod Balhorn, a biochemist at Lawrence Livermore (CA) National Laboratory. Balhorn sees two basic problems:

1. Significant unresolved interactions still exist between the scanning tip of the microscope and the surface atoms or molecules being scanned. Com-



This STM *image of individual carbon atoms on a graphite surface demonstrates STM's atomic resolution capabilities. Photo by Livermore (CA) National Laboratory.*

Watch your language

Auger effect: Transition of an electron in an atom from one discrete electronic level to an ionized continuous level of the same energy.

numerical aperture (NA): A measure of a lens's capability to gather light. NA = $n \times \sin\alpha$, where n is the refractive index of the lens and α is the angle between the outer rays entering the lens and the axis.

resolution: The ability to detect two closely spaced objects as two distinct entities. For transmitted light $R = \lambda/NA$ and for reflected light $R = \lambda/2NA$, where λ is the wavelength of light and NA is numerical aperture.

secondary ion: Particle (neutral atom or molecule, secondary electron, photon, and positive or negative ion) sputtered off a sample bombarded by a primary ion.

spectroscopy: Physics concerned with interpretation of electromagnetic spectra emitted or absorbed by a substance.

Using the scanning force microscopy technique, you get high-resolution images with little sample preparation.

R&D Perspectives

plicated interactions occur in the surface structure when electrons are pumped into the orbitals of the atoms.

2. Researchers still are learning how to interpret the topographical images produced by STMs. Simple structures like carbon are readily imaged and explained. Complex fragile structures like DNA, however, are more difficult.

A principal application of the STM is the modeling of crystal surface structures. For example, Shirley Chiang at IBM's Almaden Research Center, San Jose, CA, uses an STM to determine atomic and molecular positions, which she compares to theoretical crystal structures.

Although STMs can operate in ambient environments, Chiang operates an STM in a UHV chamber so that samples can be transferred to a low-energy diffraction instrument for additional surface analyses. She also can transfer the samples to a deposition chamber and back again to observe the effects of various coating processes.

Scanning force microscopes: Scanning force microscopes, which include atomic force microscopes (AFM), scan a sharp cantilevered probe tip at atomic distances above a sample. The resultant tip force deflections from this close contact are used to describe the physical surface characteristics of the sample.

A family of scanning force microscopes has been developed that work on the principles of attractive forces, magnetic forces, electrostatic forces, thermal variations, conductive characteristics, friction forces, acoustic characteristics, and near-field optics.

For example, researchers can use a magnetic force microscope (MFM) equipped with a nickel wire cantilever tip to image the magnetization patterns of magnetic recording materials.

"The strength of the MFM technique is that you get high-resolution images with little or no sample preparation," says Dan Rugar, at IBM's Almaden Research Center.

Commercial AFMs already are available as options to the STM.

Environmental scanning electron microscopes: The ESEM eliminates two of the biggest limitations of electron microscopy: operating the sample at UHV and having to coat samples with conductive coatings. The ESEM uses a differential pumping scheme and a gaseous discharge detector to let you look at samples at up to 20 torr.

Recent upgrades on this system include addition of a sample heating stage that can heat samples to >1,000 C. This is high enough to see oxidation-reduction reactions, crystal growth, and the melting of some metals and minerals.

Samples also can be cooled to −100 C by using liquid N_2 and Peltier-effect devices.

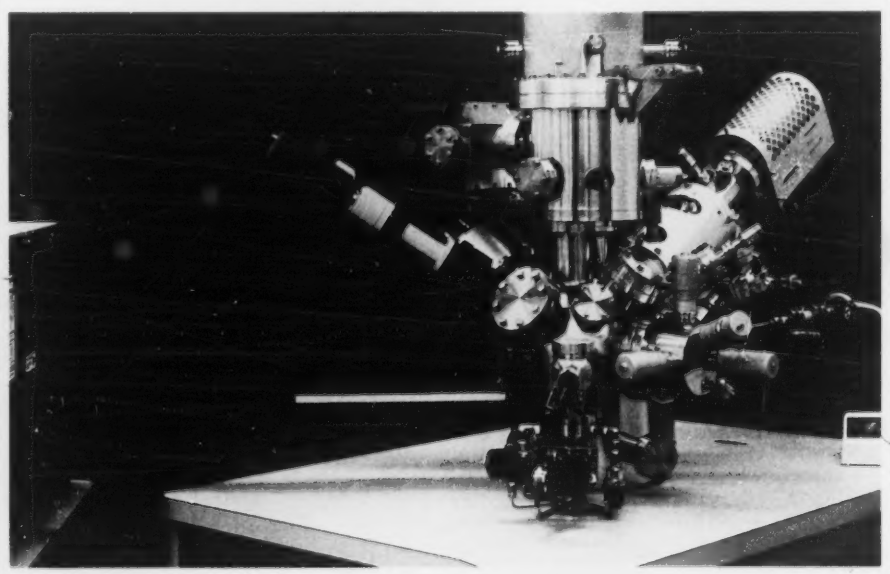
SURFACE ANALYSIS instruments are going through a maturation process that's bringing them out of the research and development stage and into use as reliable analytical tools.

"It's incredible how good some of the new instruments are now," says Charles Evans, president of the analytical service company Charles Evans & Associates, Redwood City, CA. "We have a new Auger microprobe system that hasn't been down a day in the past six months.

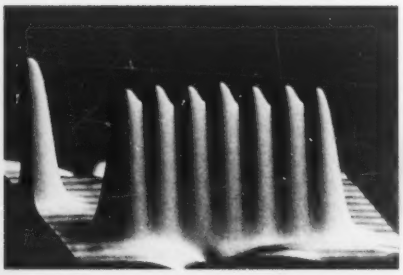
"This is such a treat compared to the maintenance required on some systems just a few years ago," he says.

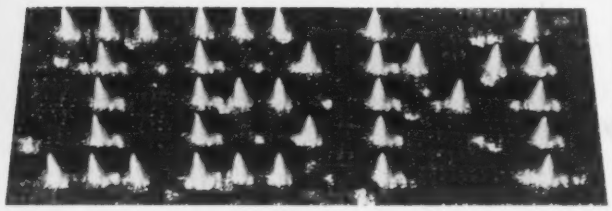
The two most popular instruments in the surface analysis field—ESCA (electron spectroscopy for chemical analysis, also known as x-ray photoelectron spectroscopy) and AES (Auger electron spectroscopy) systems—have been refined over the past several years to the point where performance is approaching theoretical detection limits.

In both ESCA and AES systems, development is centered on smaller spot sizes and more computer control, says Kenneth Bomben, XPS product manager for Perkin-Elmer, Eden



To improve sensitivity, an excimer or vacuum-UV laser ionizes sputtered species in this surface analysis by laser ionization (SALI) system .





Donald Eigler, *an IBM research physicist, is shown with the STM that he used to move and assemble individual atoms into patterns. The first cluster ever assembled (left) consisted of seven xenon atoms in a row, which was followed by a cluster of 35 xenon atoms arranged to spell "IBM" (below).*

Prairie, MN.

Future needs for materials analyses are driving the smaller spot-size requirements. "While today's materials analyses focus on material composition, future analyses also need to focus on material structure," says Robert Cormia, manager of new markets and technology at Surface Science Laboratories, Mountain View, CA.

These new instruments will need to show you the structure of the top 100 to 200 Å of a sample nondestructively. Excessive energy input into some materials can modify the structure and change the analysis results.

To do this, the current 150-μm beam diameter on an ESCA instrument will have to be reduced to 1 μm.

Although this might require a whole new ESCA design, there is plenty of incentive to accomplish this task. Biomaterials analyses, which now account for about 30% of the surface analyses performed, are expected to expand rapidly over the next five years, says George Grobe, a senior lab analyst at Perkin-Elmer, Edison, NJ.

Secondary ion mass spectrometry (SIMS), a distant third to ESCA and AES systems in the number of installed systems, also is seeing performance and design refinements.

In quadrupole-based SIMS systems, the signal often is not large enough to use the detection limits of the SIMS technique.

To offset this limitation, there is a trend toward use of static-SIMS systems to characterize the structural information of materials, with specific emphasis on organic materials.

"We've set up a new lab that only has a static SIMS capability and it is doing quite well," says Evans.

Probably the hottest area in surface analysis systems is time-of-flight SIMS (TOF-SIMS) instruments. These systems offer better sensitivity than other surface analysis systems for detection of submonolayer materials.

However, "the data from a TOF-SIMS instrument are complicated and need an expert system for materials analyses," says Cormia.

One system that seems to be catching on uses the TXRF (total reflectance x-ray fluorescence) technique. TXRF is a refinement of conventional XRF. However, instead of irradiating the sample in the traditional mode of beaming x rays down upon the surface, the x rays in a TXRF instrument are beamed at a grazing incidence to the sample.

In doing this, the x rays don't excite the bulk of the material, they excite only the near-surface region.

These systems are good for semiconductor wafers and laser optics. They have detection limits of 10^{10} atoms/cm^2.

Other surface analysis systems, such as Rutherford backscattering spectrometry, SEM-

The hottest area in surface analysis is TOF-SIMS instruments, which offer high sensitivity to detect trace elements.

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based electron diffraction x-ray, electron-probe microanalysis, and Fourier-transform infrared, also are maturing products making inroads into specific analysis areas.

"Some systems, such as ion scattering spectroscopy, are languishing," says Evans. "If they're needed, they're usually the only technique that can do the job, but often they're not really needed."

A technique that offers the potential for improved sensitivity in surface analysis is post-ionization of sputtered neutral ions. There are four ways to do this:

- Inject the sputtered neutrals into a plasma, where they're ionized by the plasma.
- Shoot an electron beam across the surface of a sample to electron impact (ionize) the sputtered ions.
- Shoot a laser tuned to a particular wavelength, across a sample's surface to ionize a specific element coming off the sample. This system is commercialized as a SARISA (surface analysis by resonance ionization of sputtered atoms) system.
- Shoot an untuned laser across a sample's surface to ionize everything. The commercialized version of this instrument is the SALI (surface analysis by laser ionization) system. To be effective, the beam-type post-ionization techniques require flat sample surfaces.

Using a different particle

Most surface analysis techniques are either mature instruments or modifications of conventional techniques.

But one new technique is the scanning proton microprobe (see accompanying figure). The SPM technique, developed at Univ. of Oxford, England, uses a beam of protons to knock out electrons. X rays, emitted as the remaining electrons rearrange themselves, are detected and

Surface analysis instruments: Determining what the surface is

	Acronym	Detection limits	Largest sample size, ϕ (cm)	Analysis time, sec	Depth of resolution, Å	Detectable elements	Approximate equipment cost (U.S. $)
Scanning Auger microscopy/spectroscopy	SAM AES	0.1 to 1.0 at-%	20	10 to 100	50*	Lithium to uranium (conductive only)	$150,000 to $500,000
Electron spectroscopy for chemical analysis	ESCA (XPS)	0.1 to 1.0 at-%	20	10 to 100	50*	Lithium to uranium (chemical groups)	$200,000 to $500,000
Secondary ion mass spectroscopy	SIMS	0.001 to 1.0 at-%	20	100	40*	Hydrogen to uranium	$200,000 to $700,000
Time-of-flight secondary ion mass spectroscopy	TOF-SIMS	<1 ppm	20	10	40	Hydrogen to uranium (large org. molecules)	$450,000 to $750,000
Energy dispersive x-ray spectroscopy (SEM acc.)	EDX	0.5 at-%	2	100	50,000	Sodium to uranium	$30,000 to $80,000
Rutherford backscattering spectroscopy	RBS	1 to 10 at-%	7	900	20,000	Lithium to uranium	$425,000 to $700,000
Ion scattering spectroscopy	ISS	1.0 at-%	20	50	10	Lithium to uranium	$150,000 to $350,000
Fourier-transform infrared spectroscopy	FTIR	1.0 at-%	7	10 to 500	10,000	Organic functional groups	$50,000 to $100,000
X-ray fluorescence spectroscopy	XRF	0.1 to 100 ppm	20	500	2×10^{8}†	Magnesium to uranium	$100,000 to $200,000
Laser ionization mass spectroscopy	LIMS	1 to 100 ppm	20	10	20,000	Hydrogen to uranium (large org. molecules)	$375,000 to $500,000
Surface analysis by laser ionization	SALI	10^{-6} of monolayer	2	50	20,000	Hydrogen to uranium	$600,000 to $800,000

* Techniques can etch in increments of 10 Å up to a few μm. † Depth of analysis depends on the matrix.

Sources: Surface Science Laboratories, Charles Evans Associates, Perkin-Elmer Corp., *R&D Magazine*

Japan, the largest single market for surface analysis systems, is buying 10 to 15 times more ESCA systems than the U.S.

R&D Perspectives

matched to material composition and structure.

While similar to XRF, SPM is more sensitive because there is very little background of unwanted x rays from other processes. Therefore, low intensity x-ray signals from low element concentrations are not obscured.

In addition to producing x rays from a sample, some of the protons bombarding the sample undergo nuclear collisions, which causes the proton itself to be backscattered out of the sample. By measuring the energy of these backscattered protons, you can measure the depth in the sample at which the collision occurred, as well as determine the nucleus involved in the collision. This means that the concentration of the elements as a function of depth into the sample can be measured.

This opens up the possibility of obtaining three-dimensional images of structures within biological cells.

Expanding foreign markets

Despite the great promise that these surface analysis techniques hold, U.S. and European researchers seem to be ignoring them. Japan currently is the largest single market for surface analysis systems.

For example, while Philips and Siemens each have about four SIMS systems, and IBM has seven, Toshiba has 15 SIMS systems, according to Stefan Daiser, ion beam systems manager for Perkin-Elmer, Eden Prairie, MN.

Japanese companies also are buying five to 10 times more ESCA systems than U.S. companies are. The only area where the U.S. still dominates is in computational-intensive Auger systems. **R&D**

READER RESPONSE

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Microscopy and surface analysis business highlights

Electron microscopes sell

ABOUT 935 scanning electron microscopes and 45 transmission electron microscopes are forecast to be sold in the U.S. and Europe in 1990, according to a survey by Strategic Directions Intl., a Los Angeles-based analytical-instrument consulting firm.

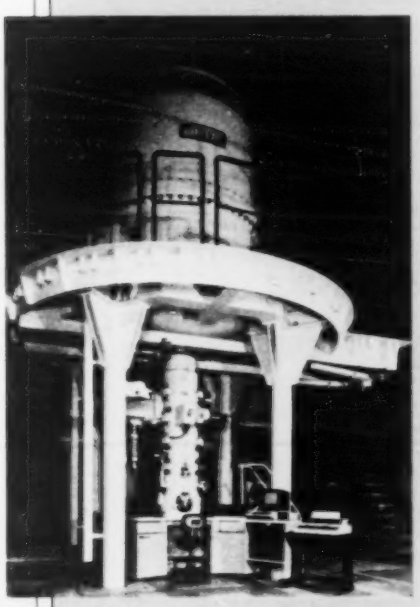
Only 50% of the SEMs in the U.S. and Europe are used in R&D applications. "SEMs are much less R&D oriented, with more systems used in quality control areas," says Lynwood Eikrem, chairman of SDI.

Japan has more patents

JAPAN, with 52 microscopy patents, edged out the U.S., with 50, for the most number of microscopy-based U.S. patents in 1989. West Germany had 17 patents. All other countries combined had 11 (19 patents were unassigned with no country of origin).

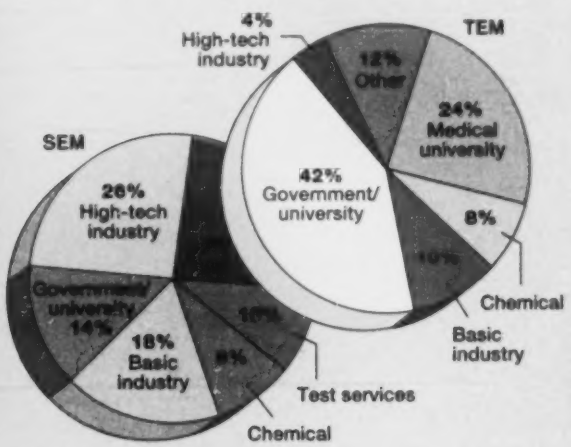
Organizations with the most microscopy patents were Olympus (12), Hitachi (11), JEOL (10), and U.S. universities (14).

For more information: MicroPatent 800/648-6787.



This 1-MV TEM at the National Center for Electron Microscopy, Berkeley, CA, has a resolution of 1.4 Å and can tilt specimens ±40 deg.

Where electron microscopes are in the U.S.



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